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(19) (CA) APPLICATION FOR CANADIAN PATENT (12)

(54) Process for Deinking Wastepaper with Fatty Acid Composition

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(71) Henkel Canada Limited - Canada ;

(57) 19 Claims

*iodine value 0-140  
millet, flakes or bread*

Notice: This application is as filed and may therefore contain an incomplete specification.

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Abstract of the Disclosure

Method and apparatus for deinking wastepaper using a fatty acid composition in molten or solid form containing a fatty acid and a nonionic surfactant, said fatty acid composition being  
5 added directly to the pulper.

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PROCESS FOR DEINKING WASTEPAPER  
WITH FATTY ACID COMPOSITION

BACKGROUND

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Related Application

This application is related to International Patent Application No. PCT/US94/06642 filed on 15 June 1994.

1.0 Field of the Invention:

10 The present invention is generally directed to a process for deinking printed wastepaper in which a fatty acid composition containing a fatty acid in molten or solid form, having a relatively low Iodine Value and a nonionic surfactant, is added to an aqueous slurry of the printed wastepaper prior to or after the addition of an alkali metal hydroxide.

15 2.0 Discussion of Related Art:

Wastepaper is used in large quantities for the production of, for example, newsprint. Lightness and color are important quality features for papers of this type. To achieve these desirable features, the printing inks have to be removed from the printed wastepaper. This is normally done by deinking processes which are generally conducted in two steps:

- 20
1. refining the printed wastepaper, i.e. fiberizing the printed wastepaper in water in the presence of the chemicals required for detachment of the printing ink particles; and
- 25

2. removing the detached printing ink particles from the fiber suspension.

The second step can be carried out by washing or flotation [Ullmanns Encyclopädie der technischen Chemie, 4th Edition, Vol. 17, pp. 570-571 (1979)]. In flotation, which utilizes the difference in watability between printing inks and paper fibers, air is forced or drawn through the fiber suspension. Small air bubbles attach themselves to the printing ink particles and form a froth at the surface of the water which is removed by savers.

The deinking of printed wastepaper is normally carried out at alkaline pH values in the presence of alkali metal hydroxides, alkali metal silicates, oxidative bleaches and surfactants at temperatures in the range from 30 to 50°C. Soap solutions are often used to detach and separate the printing inks [Ullmanns Encyclopädie der technischen Chemie, 4th Edition, Vol. 17, pp. 571-572 (1979)].

The soap solution is conventionally formed via a soap make-down system (i.e. by premixing a fatty acid composition, heated to a temperature of at least 60°C so that it is in liquid or molten form, with an alkali metal hydroxide in an amount sufficient to convert the fatty acid to soap). The soap is present in an aqueous solution in a concentration of from about 5% to about 10% by weight. If the concentration of soap significantly exceeds 10% by weight, the solution tends to form a gel which hinders the deinking process. Use of a soap solution is essential to conventional deinking processes. The soap solution is water soluble and effectively removes the ink from the paper fibers, disperses the ink into the aqueous medium and

floats the ink to the top of the aqueous medium where the ink can readily be removed from the deinking vessel (i.e. flotation cell).

While soap solutions effectively deink printed wastepaper, their preparation from a substantially pure molten fatty acid composition before entry into the pulper is time consuming and adds significantly to the overall cost of the deinking process.

It would be a significant advantage in processes for deinking printed wastepaper if a fatty acid composition could be added directly to the pulper and then combined with the alkali metal hydroxide in the pulper. This would eliminate the conventional step of converting the molten fatty acid to an aqueous soap solution in a separate vessel.

However, the addition of the molten fatty acid composition direct to the pulper presents significant problems. The fatty acid composition generally has a melting point higher than the temperature of the aqueous slurry of the printed wastepaper. As a consequence, when a molten fatty acid composition enters the pulper it congeals into a solid mass having a much lower surface area than the molten starting material. The solid mass does not readily combine with an alkali metal hydroxide (e.g. sodium hydroxide) present within the pulper and therefore does not readily form a soap solution. Thus, the deinking of printed wastepaper using substantially pure molten fatty acid without a soap make-down system has not been commercially successful.

In accordance with co-pending International Application No. PCT/US94/06642 filed on 15 June 1994, there is disclosed a process for deinking printed newspaper in which a slurry of

printed wastepaper is treated directly with a molten fatty acid in the form of fine droplets and having a low Iodine Value. The molten fatty acid readily reacts with an alkali metal hydroxide to form a soap solution which is particularly adapted for removing ink from the printed wastepaper.

It has been found that enhanced operation of the deinking process can be obtained by combining a fatty acid with a non-ionic surfactant to provide a molten or solid fatty acid composition which is added directly to the pulper. The present composition simplifies the deinking process by reducing the handling of chemicals.

### 3.0 Summary of the Invention:

The present invention is generally directed to a process for deinking printed wastepaper in which a molten or solid fatty acid composition including at least a fatty acid and a nonionic surfactant is added directly to the pulper. The present invention eliminates the step of converting the fatty acid composition into a soap solution prior to its entry into the pulper and facilitates the handling of additives employed in the deinking process.

In particular, the present invention is directed to a process for deinking printed wastepaper comprising treating a slurry of printed wastepaper with a deinking effective amount of a molten or solid fatty acid having an Iodine Value of from about 0 to 140 and a nonionic surfactant.

### Brief Description of the Drawing

Figure 1 is illustrative of an embodiment of the invention and is not intended to limit the invention as encompassed by the claims forming part of the application.

Figure 1 is a schematic view of one embodiment of the invention for deinking printed wastepaper in which a molten fatty acid composition containing a substantially pure molten fatty acid and a nonionic surfactant is fed through at least one nozzle which atomizes the molten composition into a mist of fine droplets and injects the same directly into the pulper.

### Detailed Description of the Invention

The present invention provides for the direct addition of a fatty acid composition containing a fatty acid in molten or solid form and a nonionic surfactant into a pulper to deink printed wastepaper. In accordance with the invention, the fatty acid composition is preferably in molten form and is added to the pulper as a mist of fine droplets collectively having a large surface area. The fine droplets of the fatty acid composition therefore readily react with an alkali metal hydroxide to form a soap solution. The resulting solution is particularly adapted for removing ink from the printed wastepaper.

Referring to Figure 1, the principal components employed to carry out the present invention include a storage facility 2 for storing the fatty acid composition. The storage facility 2 may be in the form of a tank for storing the molten fatty acid composition or other facility for storing the fatty acid composition in solid form. For example, the solid fatty acid

composition can be in the form of flakes or beads which can be fed directly to the pulper via bags or supersacs. The principal components further include a feed pump 4 for transferring the molten fatty acid composition to a pulper, a filter 6 for removing contaminants from the molten fatty acid composition to produce a substantially pure feed, a flowmeter 8 for metering the molten fatty acid feed to the pulper, at least one nozzle 10 for converting the molten fatty acid feed into a mist of fine droplets and a pulper 12 for combining the mist of molten fatty acid with an aqueous slurry containing the printed wastepaper.

A fatty acid composition is fed from a source (not shown) via a conduit 14 to the storage tank 2. The fatty acid composition which may be employed in the present invention includes fatty acids having a relatively high degree of saturation as measured by the Iodine Value. The fatty acids have an Iodine Value generally in the range of from 0 to about 140, preferably from 0 to about 80. The preferred fatty acids include straight chain fatty acids having from 8 to 22 carbon atoms and mixtures thereof.

The fatty acid component of the deinking composition is present in an effective amount sufficient to form a soap solution to remove ink from the wastepaper. Preferably, the amount of the fatty acid component is from about 30 to 95% by weight based on the total weight of the fatty acid composition.

The present fatty acid composition also includes at least one nonionic surfactant. The preferred groups of nonionic surfactants include alkyl polyglycosides, alkoxylated  $C_8 - C_{20}$  alcohols, alkoxylated  $C_8 - C_{22}$  fatty acids and alkoxylated mono-



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di- and triglycerides. The nonionic surfactants are preferably present in amounts of from about 5 to 70% by weight based on the total weight of the composition.

The preferred alkyl polyglycosides include APG 625 Glycoside, a nonionic alkyl polyglycoside composition sold by  
5 Henkel Canada Ltd. The preferred alkoxyated  $C_8 - C_{20}$  alcohols include  $C_{16} - C_{18}$  alcohols. The preferred alkoxyated  $C_8 - C_{22}$  fatty acid is palmitic acid. The preferred alkoxyated glycerides include Peratom 853, an ethoxylated vegetable oil sold  
0 by Henkel Corporation.

The molten fatty acid composition may also include at least one additive selected from the group consisting of cellulosic derivatives and complexing agents.

The cellulosic derivatives are added to improve ink  
5 dispersion in the pulper and for preventing small ink particles from redepositing on the fiber. The amount of the cellulosic derivatives is typically up to about 5% by weight, based on the total weight of the composition. Preferred examples of the cellulosic derivatives include carboxymethyl cellulose,  
0 hydroxyethyl cellulose and the like and mixtures thereof. The cellulosic derivatives are present in an amount of up to about 5% by weight based on the total weight of the fatty acid composition.

Complexing agents are also preferably used to chelate heavy  
5 metal ions such as copper, chromium, iron and the like. In the absence of complexing agents, the heavy metals may decompose the hydrogen peroxide contained in the pulper. The preferred complexing agents include diethylenetriamine pentaacetic acid,

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diethylenetriamine pentamethoxyphosphonic acid, their salts and the like, and mixtures thereof. The complexing agents are present in an amount of up to about 10% by weight based on the total weight of the fatty acid composition.

5           The fatty acid composition of the present invention can be maintained in the molten state by applying heat such as with a heating coil 18 which receives steam via a conduit 16 and removes condensate via a line 20. The molten fatty acid composition, heated to a temperature above its titre (melting point),  
10 preferably to a temperature of at least 60°C, most preferably about 70°C, moves from the tank 2 via a conduit 22 to the feed pump 4 where pressure is applied to generate a feed stream having a constant flowrate of from about 5 liters/minute to 25 liters/minute.

15           The pressurized feed is then sent via a conduit 24 to the filter 6 which improves the purity of the fatty acid composition by removing contaminants such as carbon particles and the like. The resulting purified feed stream is divided into a recycled stream which flows back to the tank 2 via a conduit 26 and a  
20 valve 28 and a process stream which flows via a conduit 30 and valve 32 to a flowmeter 8. The conduit 30 is preferably purged with steam after the passage of each charge of the molten fatty acid composition. The steam which is provided from a source (not shown) through a conduit 34 and a valve 36, removes residual  
25 amounts of the fatty acid composition. In addition, the steam preheats the conduit 30 to operation temperatures (i.e. above the titre of the fatty acid composition) to insure passage of the fatty acid composition in molten form.

( The process stream then passes through flowmeter 8 which controls the rate at which the purified molten fatty acid composition is sent via a conduit 38 to the nozzle 10. The flowmeter 8, which is conventional in the art, regulates the flow of the process stream on a volume or mass basis. The flowmeter 8 registers the amount of the fatty acid composition which passes into the pulper until a predetermined amount is reached and then the flowmeter shuts off thereby terminating the flow of the fatty acid composition into the pulper.

The metered process stream containing the purified molten fatty acid composition is then sent to at least one injection nozzle which atomizes the molten fatty acid composition into a mist of fine droplets cumulatively having a very large surface area. The large surface area enables the molten fatty acid composition to more readily react with the alkali metal hydroxide in the pulper 12 to form a soap solution.

The size of the droplets of the fatty acid composition obtained from the nozzle should be sufficient to promote a reaction with the alkali metal hydroxide. It is desirable for the fine droplets to have a typical particle size of from about 200 to 900 microns, preferably about 300 to 500 microns. Particles of this size can readily be produced from conventional nozzles.

The mist of the molten fatty acid composition is sprayed from the nozzle 10 into the pulper 12 which contains an aqueous solution of deinking chemicals (e.g. sodium silicate, sodium hydroxide and hydrogen peroxide) as described hereinafter. The process water for the aqueous solution is provided through a

conduit 40 while the deinking chemicals are provided through a conduit 42. The printed wastepaper to be deinked enters the pulper 12 via a conveyor 44 and is present in a concentration typically in the range of from about 4 to 20% by weight, more typically 10 to 14% by weight.

As previously indicated, if the fatty acid composition is in solid form (e.g. flakes, beads and the like), the composition can be introduced directly into the pulper 12 through the use of conventional bags or supersacs.

The printed wastepaper is refined at a temperature of from 20 to 60°C in the aqueous slurry typically containing as deinking chemicals from about 0.5 to 3% by weight of each of hydrogen peroxide, sodium silicate (35% by weight 37 - 40°Bé) and an alkali metal hydroxide, preferably sodium hydroxide, based on the weight of the printed wastepaper. The residence time of the printed wastepaper in the pulper during the deinking process is from about 10 to 30 minutes. The conditioned pulp is removed from the pulper via the conduit 46.

The type of ink which may be removed from the printed wastepaper in accordance with the present invention may be oil-based or water-based. For water-based inks, the aqueous slurry of the wastepaper may also contain water-soluble polymers and/or copolymers containing amino and/or ammonium groups such as disclosed in United States Patent 5,286,346 issued February, 15, 1994, incorporated herein by reference.

The following examples are illustrative of embodiments of the invention and are not intended to limit the invention as encompassed by the claims forming part of the application.

EXAMPLE 1

20 tons of a molten fatty acid composition having an Iodine Value of 14, containing about 7% by weight of a blend of principally  $C_{14}$  -  $C_{20}$  ethoxylated fatty acids (nonionic surfactant) and a titre (melting point) of 52°C (Olinor 4013-CA produced by Henkel Canada Limited) was kept at 65 - 70°C in a 30 ton bulk storage tank equipped with a heating coil. The material was continuously circulated through a 1 inch insulated and steam-traced recycle line equipped with a feed pump and an in-line bag filter. A one inch spray line was branched off from the recycle line and positioned as closely as possible to the pulper.

A batch pulper capable of handling 8.6 ADMT (Air Dried Metric Tons) of printed wastepaper at 12% consistency was equipped with a set of 4 spraying nozzles having the following specifications: Fulljet nozzle spray, female, 3/8 inch, Cat. No. 3/8 GS9.5 (John Brooks Company Ltd., Mississauga, Ontario L4W 1Y1).

The pulper was first charged with 55 tonnes of recirculated White Water at 45°C and followed by:

- (1) 90 liters of sodium hydroxide (50%);
- (2) 86 Kg of sodium silicate (40%  $Na_2Si_2O_5$ );
- (3) 7 liters of DTPA (diethylenetriamine pentaacetic acid, 40%); and
- (4) 130 liters of hydrogen peroxide (50%).

The fatty acid composition spray line was first steam purged through a micromotion mass flowmeter all the way to the nozzle heads for one (1) minute or until the line was hot and clear. While the pulper agitator was on, the fatty acid valve at the

tee-off position of the spray line was opened while the valve at the recycle line was gradually closed to build up a line pressure of 60 psi. The molten fatty acid composition was metered through the flowmeter and sprayed directly into the contents of the pulper in fine droplets having a typical particle size of from about  $212\mu$  to  $850\mu$ . After a total of 66 kg of the molten fatty acid composition was registered by the mass flowmeter in 5-minute spraying time, the fatty acid valve on the recycle line was opened and the valve on the spray line was closed. Without delay, the spray line was purged for one minute with 120 pound steam to clean the line and nozzles. This completed the batch cycle of the fatty acid spraying application.

As soon as the fatty acid composition droplets hit the alkaline White Water, they were saponified/dissolved instantaneously into a water soluble solution without forming lumps of unreactive fatty acid. This solution, having a concentration of the fatty acid composition of about 0.12% by weight, acted as a surfactant for the detaching/dispersing of the ink particles from the 8.6 ADMT wastepaper which was introduced to the pulper during or after the addition of the fatty acid composition. Eventually, in the presence of hard water, the fatty acid composition in its water-soluble form agglomerated and floated the ink particles to the surface in its final active chemical form as a precipitated calcium soap.

In an alternate embodiment of the invention, the alkali metal hydroxide is added to the pulper after the fine mist of the molten fatty acid composition is injected into the pulper. When the molten fatty acid composition enters the pulper, it cools and

forms small, solid microbeads. The surface area of the microbeads is sufficiently large so that substantially all of the fatty acid reacts with the subsequently added alkali metal hydroxide to form the soap solution necessary to deink the printed wastepaper.

#### EXAMPLE 2

A batch pulper capable of handling 8.6 ADMT of printed wastepaper at 12% consistency was first charged with 55 tonnes of recirculated White Water at 45°C and followed by:

- (1) 90 liters of sodium hydroxide (50%);
- (2) 86 Kg of sodium silicate (40% B<sub>2</sub>);
- (3) 7 liters of DTPA (diethylenetriamine pentaacetic acid, 40%); and
- (4) 130 liters of hydrogen peroxide (50%).

Prior to the addition of 8.6 ADMT of printed wastepaper, 66 Kg of the fatty acid composition identified in Example 1 in the form of microbeads was charged to the pulper.

As soon as the fatty acid composition microbeads hit the alkaline White Water, they were saponified instantaneously into a water soluble solution without forming lumps of unreactive fatty acid. This solution, having a concentration of the fatty acid composition of about 0.12% by weight, acted as a surfactant for the detaching/dispersing of the ink particles from the fiber. Eventually in the presence of hard water, the fatty acid composition agglomerated and floated the ink particles to the surface in its final active chemical form as a precipitated calcium soap.

What is claimed is:

1. A process for deinking printed wastepaper comprising treating a slurry of printed wastepaper with a deinking effecting amount of a fatty acid composition, said fatty acid composition comprising:

- (a) a fatty acid having an Iodine Value of from about 0 to about 140; and
- (b) at least one nonionic surfactant.

2. The process of claim 1 wherein the fatty acid is in molten form.

3. The process of claim 2 wherein the molten fatty acid is in the form of fine droplets.

4. The process of claim 1 wherein the fatty acid is in solid form.

5. The process of claim 4 wherein the fatty acid is a solid in the form of flakes or beads.

6. The process of claim 1 wherein the amount of the fatty acid is from about 95 to 30% by weight and the amount of the nonionic surfactant is from about 5 to 70% by weight, based on the total weight of the composition.



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7. The process of claim 1 wherein the fatty acid is a mixture of straight chain fatty acids having from 8 to 22 carbon atoms.

5 8. The process of claim 1 wherein the nonionic surfactant is selected from the group consisting of alkyl polyglycosides, alkoxyated C<sub>8</sub> - C<sub>20</sub> alcohols, alkoxyated C<sub>8</sub> - C<sub>22</sub> fatty acids and alkoxyated mono-, di- and triglycerides.

10 9. The process of claim 1 wherein the fatty acid composition further comprises an effective amount of at least one cellulosic derivative.

10. The process of claim 9 wherein the cellulosic derivative is present in an amount of up to about 5% by weight, based on the total weight of the composition.

15 11. The process of claim 9 wherein the cellulosic derivative is selected from the group consisting of carboxymethyl cellulose and hydroxyethyl cellulose.

12. The process of claim 1 wherein the fatty acid composition further comprises an effective amount of at least one complexing agent.

20 13. The process of claim 12 wherein the complexing agent is present in an amount up to about 10% by weight, based on the total weight of the composition.

14. The process of claim 12 wherein the complexing agent is selected from the group consisting of diethylenetriamine pentaacetic acid, diethylenetriamine pentamethoxyphosphonic acid and salts thereof.

15. The process of claim 1 wherein the fatty acid has an Iodine Value of from about 0 to 80.

16. The process of claim 3 wherein the fine droplets have a particle size of from about 200 to 900 microns.

17. The process of claim 1 wherein the slurry of printed wastepaper comprises deinking chemicals including sodium silicate, hydrogen peroxide and a surfactant.

18. The process of claim 17 wherein said deinking chemicals further comprise an alkali metal hydroxide.

19. The process of claim 18 comprising adding the fatty acid composition to the aqueous solution of deinking chemicals to form a mixture, and adding the printed wastepaper to the mixture to form said slurry.

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